Extended Abstract: NRIP 1997

The NIST Radiochemistry Intercomparison Program NRIP: Traceability for Low-Level Radioanalytical Laboratories-Results of Year 1.

Michael K. Schultz, Kenneth G. W. Inn, J. M. R. Hutchinson. National Institute of Standards and Technology, Ionizing Radiation Division, Building 245/Room C114, Gaithersburg, MD, 20899. Phone (301) 975-4336; Fax (301) 926-7416; email: michael.schultz@nist.gov

## Introduction

The National Institute of Standards and Technology NIST together with participating laboratories (Table 1) has established a traceability-testing program for low-level radioactivity measurements. Three rounds of testing were completed in year 1. The matrices and activity concentrations reflected common radioanalytical analyses performed by the participating laboratories. Test materials include both environmental (water, soils, sediments, vegetation, etc.) and bioassay (urine, fecal material) sample matrices. Laboratories were issued Certificates of Traceability for the matrices and nuclides of the tests. The program has been implemented to meet the demand for traceability as defined under ANSI N42.23. This voluntary standard defines a hierarchy of traceability with an unbroken linkage to NIST (Figure 1). The system is configured to include a small number of so-called reference laboratories which are to act as performance evaluation PE materials providers to both monitoring and service laboratories. The standard further envisions the exchange of testing materials between the reference laboratories and NIST. Under the standard, the testing materials are (1) to be composed of appropriate matrices (i.e., matrix categories commonly analyzed by the laboratory) and (2) to consist of appropriate (commonly encountered) activity concentration ranges for these matrices. Currently however, no such testing exists between NIST and laboratories providing performance evaluation materials at environmental levels. Consequently, it has been necessary for laboratories in need of traceability for low-level natural-matrix and bioassay sample measurements to seek direct traceability testing with NIST.

## Test Results for Year 1

The first year of testing consisted of three test-sample distributions. Each distribution comprised 5 test samples and 3 blank samples for analysis. Test samples were spiked with <sup>238</sup>Pu, <sup>239/240</sup>Pu, and <sup>241</sup>Am (included as alpha-energy interference for <sup>238</sup>Pu). Activity concentration range for all tests was 2-20 decays per minute (dpm) per sample. The sample matrices were aqueous (diluted nitric acid solution) (Round 1), glass-fiber filters (Round 2), and artificial urine (Round 3). A total of 8 laboratories participated in the exercises during year 1 (Table 1). Results are presented here for test-sample distributions 1 and 2 (Figs. 2-7).

## Data Analysis/ Acceptance Criteria

The program has evolved over the course of the year to include acceptance criteria for submitted results. In an effort to conform traceability testing by NIST to industry standards, the criteria for acceptance of measurements for traceability to NIST were derived in the spirit of ANSI N42.22. ANSI 42.22 defines the acceptance criteria for traceability and a traceability limit to NIST for performance testing as:

$$\left|V_{NIST} - V_{Rpt}\right| \le 3 \times \sqrt{\sigma_{NIST}^2 + \sigma_{Rpt}^2}$$

Where:  $V_{NIST}$  and  $V_{Rpt}$  are the NIST value and the laboratory reported value respectively;  $\sigma_{NIST}$  and  $\sigma_{Rpt}$  are the NIST and reported uncertainties; and the quantity "3 times the square root of the combined sum of the squares of the NIST and reported uncertainties" represents the traceability limit. These criteria were applied for Test 2 and included in the Report of Traceability provided to the individual participants following testing and data analysis.

## Future Testing

The distribution dates and sample matrices for fiscal year 1998 were decided at the 1st NRIP users meeting held August 19-21 in Carlsbad, NM. Matrices for the coming year are aqueous solutions, glass-fiber filters, artificial urine and fecal material, and soil. The analyte list has been expanded to include <sup>238</sup>Pu, <sup>239</sup>Lu, <sup>234</sup>U, <sup>90</sup>Sr. Distributions are to begin in the first week of December 1997.

Extended Abstract: NRIP 1997

Table 1 List of participating laboratories and contact individuals for NRIP 1997 traceability testing.

Organization	Abbreviation	Contact(s)	Test Participation (Year 1)
Auburn University	AU	Jon Broadway	1,2
Carlsbad Environmental Monitoring and Research Center	CEMRC	Shan Lee	1,2,3
Environmental Evaluation Group	EEG	Donald Gray	1,2,3
National Air and Radiation Environmental Laboratory	NAREL	John Griggs James Moore	1,2,3
Oak Ridge National Laboratory	ORNL	Myint Thein	3
Sandia National Laboratory	SNL	Amir Mohagheghi Frank Loudermilk	1,2,3
Westinghouse Savannah River Company	WSRC	Brian Crandall Robert Henderson	1,2,3
Westinghouse Waste Isolation Pilot Plant	WIPP	Steven Bakhtiar	1,2,3

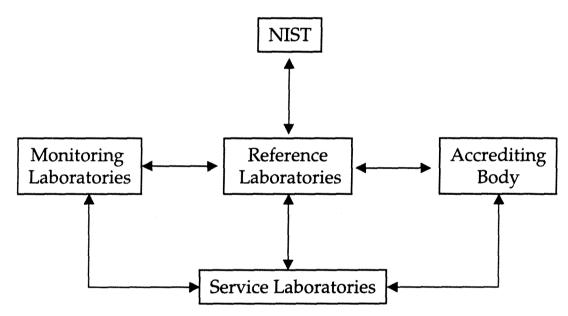


Figure 1 Traceability testing and accreditation process envisioned in ANSI N42.23. Arrows between laboratories represent the exchange of test samples for performance evaluation testing. An accrediting body (not yet in place) would serve to assure that the testing process was complete and adequate. Reference Laboratories must have the capability of preparing and distributing test samples to both Monitoring and Service Laboratories. The traceability testing of measurements is only a part of the total accreditation process. Also needed are internal QA/AC and On-site assessments.

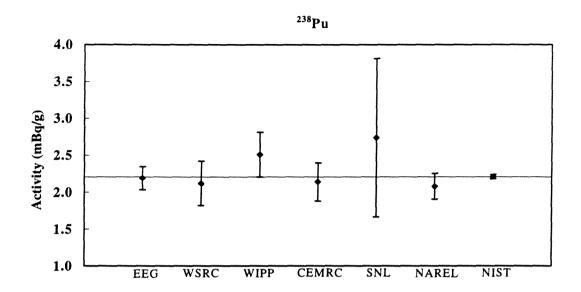


Figure 2 Test results for <sup>238</sup>Pu for NRIP397 (Test 1, actinides in nitric acid solution). Grand mean for reported results (excluding NIST),  $\mu$ = 2.30±0.53 (23%, 2 sigma) mBq•g<sup>-1</sup>; % bias versus NIST value = +3.9%. Error bars represent a two standard deviation of the mean interval as reported by individual laboratories. For Laboratory codes, see table 1.

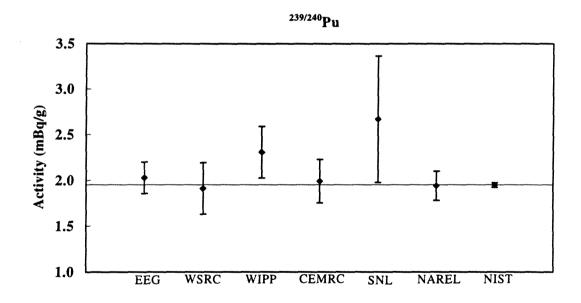


Figure 3 Test results for  $^{239/240}$ Pu for NRIP397 (Test 1, actinides in nitric acid solution). Grand mean for reported results (excluding NIST),  $\mu$ = 2.14±0.59 (28%, 2 sigma) mBq•g<sup>-1</sup>; % bias versus NIST value = +9.8%. Error bars represent a two standard deviation of the mean interval as reported by individual laboratories. See Figure 1 for laboratory codes.

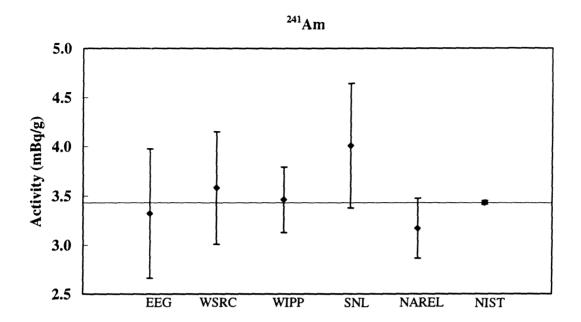


Figure 4 Test results for  $^{241}$ Am for NRIP397 (Test 1, actinides in nitric acid solution). Grand mean for reported results (excluding NIST),  $\mu$ = 3.51±0.64 (18%, 2 sigma) mBq•g<sup>-1</sup>; % bias versus NIST value = +2.27%. Error bars represent a two standard deviation of the mean interval as reported by individual laboratories.

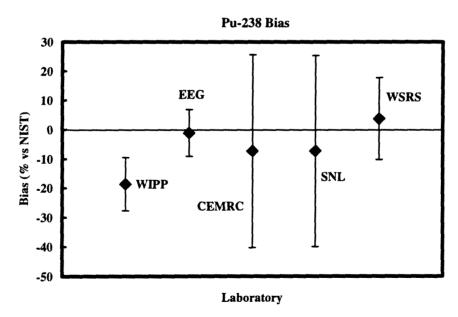


Figure 5 Mean bias of reported <sup>238</sup>Pu data for NRIP 6/97 (Test 2, actinides on glass-fiber filters). Horizontal line represents the NIST value. Uncertainties are quoted at a 2 sigma level, representing an estimate of a 95% confidence interval for the mean bias.

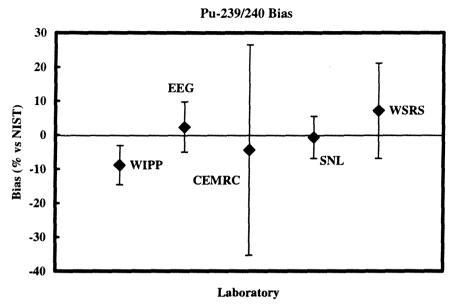


Figure 6 Mean bias of reported <sup>239/240</sup>Pu data for NRIP 6/97 (Test 2, actinides on glass-fiber filters). Horizontal line represents the NIST value. Uncertainties are quoted at a 2 sigma level, representing an estimate of a 95% confidence interval for the mean bias.

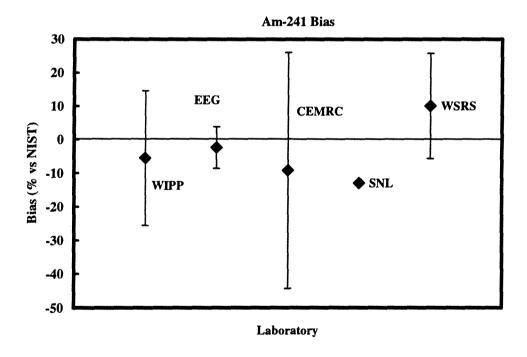


Figure 7 Mean bias of reported <sup>241</sup>Am data for NRIP 6/97 (Test 2, actinides on glass-fiber filters). Horizontal line represents the NIST value. Uncertainties are quoted at a 2 sigma level, representing an estimate of a 95% confidence interval for the mean bias.